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## High-resolution Powder Diffraction Studies of Sulfate-attacked Portland Cement

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Beamline(s): X14A

**Introduction:** Powder diffraction data were collected on beamline X14A as part of a program of research investigating the mechanisms of sulfate attack on Portland cement. The data were intended to compliment results already obtained using spatially-resolved energy-dispersive x-ray diffraction (EDXRD) at the APS. In particular, the aim was to overcome the limited reciprocal-space resolution inherent in the EDXRD method, and to facilitate identification of the somewhat complex mixtures of crystalline phases contained in the sulfate-attacked cement samples.

**Methods and Materials:** Samples of ordinary Portland cement paste were cast in the form of cylinders 12 mm in diameter and 40 mm in length. They were exposed to  $\text{Na}_2\text{SO}_4$  and  $\text{MgSO}_4$  solutions (equivalent to 33,800 ppm of  $\text{SO}_4$ ) for sixteen weeks. From each treated cylinder, three powder samples were carefully extracted:

1. From the surface layer, *i.e.* up to 1 mm below the surface
2. From the region 1–4 mm below the surface
3. From the interior of the sample.

In addition, a sample was taken from an unexposed control; there were seven powders in all. All powder preparations were performed in a dry glove box to prevent atmospheric carbonation.

While this procedure hardly approaches the degree of spatial resolution obtainable in the APS data, it is sufficient to enable some comparisons to be made between the phase compositions of the attacked cement pastes at different depths below their exposed surfaces. The amounts of powder available for each data collection were necessarily small, so samples were loaded into capillaries and Debye-Scherrer geometry was used, with an incident beam wavelength of 1.24 Å.

Data were also collected from samples of unreacted cement powder; one from the same type I cement used to prepare the sulfate-attack samples, and one of a type V or “sulfate-resisting” cement.

**Results:** The data obtained enabled the presence (in abundance) of several phases which had not been readily observable in the APS data, although not all of these have proved to be easily identifiable. In particular, the presence of ettringite and so-called ‘monosulfate’ in several distinct hydration states was established. A clear differentiation was observed between powder samples from surface layers in both  $\text{Na}_2\text{SO}_4$ - and  $\text{MgSO}_4$ -exposed samples. The surface layer of the  $\text{Na}_2\text{SO}_4$ -exposed sample contained a phase which has been tentatively identified as ‘sodium pentasalt’,  $\text{Na}_2\text{SO}_4 \cdot 5\text{CaSO}_4 \cdot 3\text{H}_2\text{O}$ ; this phase has not previously been associated with sulfate attack. The  $\text{MgSO}_4$ -exposed sample surface layer contained a phase giving heavily broadened reflections, possibly due to disordering along one crystallographic axis. This phase has not been identified.

**Conclusions:** Examination of the high-resolution powder data has led to extensive and successful modification of our data collection strategy at the APS, such that in data from more recent visit we have been able to obtain a wealth of phase information from our EDXRD data while maintaining a high degree of spatial resolution.

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